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PRELIMINARY STUDY OF OXIDE-DISPERSION-STRENGTHENED B-1900 PREPARED BY MECHANICAL ALLOYING

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16. Abstract

An experimental oxide-dispersion-strengthened (ODS) alloy based on the B-1900 composition was produced by the mechanical alloying process. Without optimization of the processing for the alloy or the alloy for the processing, recrystallization of the extruded product to large elongated grains was achieved. Materials having grain length-width ratios of 3 and 5.5 were tested in tension and stress-rupture. The ODS B-1900 exhibited tensile strength similar to that of cast B-1900. Its stress-rupture life was lower than that of cast B-1900 at 760° C. At 1095° C the ODS B-1900 with the higher grain length-width ratio (5.5) had stress-rupture life superior to that of cast B-1900. It was concluded that, with optimization, oxide dispersion strengthening of B-1900 and other complex cast nickel-base alloys has potential for improving high-temperature properties over those of the cast alloy counterparts.

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PRELIMINARY STUDY OF OXIDE-DISPERSION-STRENGTHENED B-1900 PREPARED BY MECHANICAL ALLOYING

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SUMMARY

The objective of this preliminary study was to determine whether a complex superalloy, normally used in the cast condition for gas turbine blade application, could be dispersion strengthened by the mechanical alloying process. Evaluation of the potential for dispersion strengthening was made primarily in terms of the development of a fine oxide dispersion and the achievement of large elongated grains.

The superalloy B-1900 was chosen as representative of complex cast alloys used for gas turbine blades. Experimental B-1900 to which 1 volume percent of yttrium oxide was added was prepared for this study by the International Nickel Company, Inc., by the mechanical alloying process. Neither the alloy nor the process was varied to achieve optimum results.

Mechanically alloyed powder was consolidated by extrusion. The product, examined after heat treatment, exhibited a fine oxide dispersion and somewhat elongated large grains. Grain aspect ratios (average grain length-width ratios) of 3 and 5.5 were observed. The recrystallized oxide-dispersion-strengthened (ODS) B-1900 was tested in tension and stress-rupture. Comparisons were made with literature values for cast B-1900.

The ODS B-1900 and cast B-1900 exhibited similar ultimate tensile strengths, with ODS B-1900 having the advantage at low temperature and cast B-1900 having the advantage at high temperature. The yield strength of ODS B-1900 exceeded that of cast B-1900 from room temperature to 1095° C.

The 760° C stress-rupture life of ODS B-1900 was below that of cast B-1900. The ODS B-1900 with a grain aspect ratio of 5.5 was slightly superior in 1095° C stress-rupture life to cast B-1900, while the grain-aspect-ratio-3 ODS B-1900 was inferior. Stress-rupture life was improved at both 760° and 1095° C by increasing aspect ratio; tensile properties, however, were independent of this factor. Introduction of a notch decreased stress-rupture life at 760° C but did not affect 1095° C rupture life.

While the potential for application of the mechanical alloying technique to complex alloys was demonstrated, the need for optimization of grain aspect ratio to achieve superior properties was evident.

INTRODUCTION

Advances in the design and utilization of gas turbine engines call for ever more capable alloys. Especially demanding are the requirements for gas turbine blades: extraordinary strength at high temperatures for prolonged periods of time. Currently these needs are met by cast nickel-base superalloys such as 713-C, B-1900, and IN-100; but because use conditions will become more severe, more advanced alloys are being sought.

Oxide dispersion strengthening is one of the methods by which an improved creep resistant material may be produced. To achieve the fine oxide dispersion required for oxide dispersion strengthening the mechanical alloying process has been developed. In this process, metal and oxide powders are placed in a high-energy stirred ball mill. The most commonly used mill is the Union Process Company attritor. In the course of milling, a repeated sequence of powder consolidation, thinning, fracture, and rewelding occurs. The product powder is a highly worked composite of uniformly distributed metal and oxide components (ref. 1). This technique has been quite successfully applied to the relatively simple alloy Nimonic 80 (ref. 2) as well as to somewhat more complex alloys containing refractory elements for solid solution strength and higher levels of the γ' formers aluminum and titanium (refs. 3 and 4). Combining oxide dispersion strengthening and γ' precipitation strengthening can yield a material having both strength derived from the γ' phase at intermediate temperatures and strength derived from the oxide dispersion strengthening at higher temperatures, where γ' precipitation strengthening becomes ineffective. Thus, considerable interest exists in extending the range of alloy compositions which may be effectively dispersion strengthened and in developing dispersion-strengthened alloys with potential for turbine blade use.

The objective of this study was to determine whether a complex superalloy, normally used in the cast condition, could be effectively dispersion strengthened as judged by achievement of the following factors:

(1) A fine and uniform oxide dispersion

(2) Secondary recrystallization to large elongated grains considered necessary for high-temperature strength in oxide-dispersion-strengthened (ODS) materials

(3) Improved strength compared with that of the cast version of the alloy

Because this was a preliminary study, greater weight was given in evaluation to the quality of the oxide dispersion and to the grain size and shape achieved as showing the potential for improved strength. Correspondingly less weight was given in evaluation to the mechanical properties.

In this investigation, the mechanical alloying technique of dispersion strengthening was applied to B-1900, an alloy representative of current high-strength cast nickel-base alloys used for gas turbine blades. It is a complex alloy in that it is strengthened

by γ' ($\sim$ 60 vol.%), refractory metal solid solution hardeners, and carbides (ref. 5). The ODS B-1900 with an intentional addition of 1 volume percent yttrium oxide (Y_2O_3) was prepared by mechanical alloying at the Paul D. Merica Research Laboratory by the patented (ref. 1) International Nickel Company, Inc., process. Subsequent to processing and heat treatment, the alloy was evaluated at the Lewis Research Center. The alloy was prepared for this preliminary study on a "best effort" basis with no attempt made to alter the process to suit the alloy or vice versa.

This report discusses results of microstructural examinations and mechanical property determinations conducted with the ODS B-1900. The microstructural examinations included both electron and optical microscopy. Test specimens were machined from ODS B-1900 with two values of grain aspect ratio (average grain length-width ratio). The mechanical tests included tensile tests at temperatures ranging from room temperature to 1095° C and stress-rupture tests conducted at 760° and 1095° C. Because of the concern with the low ductility of some ODS materials, some tests were included to determine the effect of notch-induced stress concentration on tensile strength and rupture life. Comparisons were made with literature values for cast B-1900.

PROCEDURE

Preparation of Mechanically Alloyed Oxide-Dispersion-Strengthened B-1900

The materials for this study were prepared by the mechanical alloying process by the Paul D. Merica Research Laboratory of the International Nickel Company, Inc. The processing steps for mechanical alloying are described in the patent literature (refs. 1, 6, and 7). Typically these steps include attritor processing, hot consolidation, and high-temperature annealing. In attritor processing ductile less reactive elemental powders (e.g., nickel (Ni) or cobalt (Co)), brittle master alloys containing reactive elements (e.g., aluminum (Al) or titanium (Ti)), and fine stable oxide powders (e.g., Y_2O_3 or lanthanum oxide (La_2O_3)) are intimately blended in a stirred ball mill. Processing conditions are adjusted to cause a repeated sequence of powder consolidation, thinning, fracture, and rewelding to produce a highly worked composite of metal and oxide components. The blend is consolidated and worked, usually by hot extrusion at ratios varying from 12 to 1 to 24 to 1 and temperatures of the order of 1100° C. Finally, the extruded mechanically alloyed product is annealed at a more elevated temperature for recrystallization to change the very fine grains to coarse elongated grains.

The compositions of the extruded rod produced for this study and of conventional cast B-1900 (ref. 8) are given in table I. The carbon was intentionally low in the ODS

alloy to avoid formation of potentially deleterious carbide phases. After extrusion the ODS B-1900 was given a two step heat treatment consisting of 1/2 hour at 1245° C (followed by air cooling) and 24 hours at 845° C (followed by air cooling). The first step of the heat treatment was intended to cause recrystallization to coarse well elongated grains considered necessary for high-temperature strength, while the second step was intended to develop the γ' precipitate. All microstructural and mechanical property evaluations of ODS B-1900 were made in the heat-treated condition. Gradient annealing, often used to improve grain structures and mechanical properties of ODS products, was not applied to the materials discussed in this report.

Evaluation of Oxide-Dispersion Strengthened B-1900

Examination of the microstructure of recrystallized ODS B-1900 was conducted by optical and electron microscopy. An immersion etchant consisting of 2.5 grams of ferric chloride and 2.5 grams of cupric chloride in 10 cubic centimeters of nitric acid, 50 cubic centimeters of hydrochloric acid, and 30 cubic centimeters of ethyl alcohol effectively delineated the microstructure. The grain aspect ratio (GAR) was determined by an intercept method; the GAR was taken as the ratio of the average length between intersections of grain boundaries with lines parallel to the longitudinal axis to the corresponding average length in the transverse direction.

Specimens of ODS B-1900 having two different aspect ratios were used for tensile and stress-rupture tests. The two aspect ratios apparently resulted from different amounts of work (ref. 3). The specimen configurations used are shown in figure 1. Tensile tests were conducted at room temperature and 760°, 870°, 980°, and 1095° C at a strain rate of 0.02 per minute. Stress-rupture testing was conducted at 760° and 1095° C. Circumferentially notched specimens were ground to have a stress concentration factor of approximately 3; these were tested in tension and stress-rupture at 760° and 1095° C. All testing was performed in air. All cast B-1900 data used for comparison were from the literature (ref. 8); the literature data were for smooth specimens only, tested in the as-cast condition. The grain size of cast alloys such as B-1900 is usually in the range 1.5 to 3 millimeters. Specimens were examined metallographically after testing to determine fracture characteristics.

To determine the identity of the nonmetallic phases in ODS B-1900, a sample was dissolved in 10 percent bromine in methanol. The extraction residue was separated into a heavy (coarse) fraction and a light (fine) fraction by centrifugal sedimentation. These two fractions were separated in an effort to identify the usually undesirable coarser nonmetallic particles observed in the microstructure of ODS B-1900 (fig. 2). The finer fraction was recovered by evaporation of the supernatant liquid decanted after

centrifuging. The coarser fraction had settled to the bottom of the centrifuge tube. The two fractions were examined by X-ray diffraction and spectrographic analysis.

The oxygen and nitrogen contents of ODS B-1900 were determined by inert gas fusion analysis; the carbon content was determined by combustion chromatographic analysis.

RESULTS AND DISCUSSION

Microstructure of Oxide-Dispersion-Strengthened B-1900

Nature of the dispersion. - An electron microscopy replica photograph of the ODS B-1900 prepared for this study is shown in figure 2. In addition to the γ and γ' phases, the ODS B-1900 contained nonmetallic particles ranging in size from 0.006 to 0.5 micrometer (60 to 5000 Å). Most of the particles were fine and well dispersed. The fine particles occurred within both the γ and γ' phases.

The oxygen content of extruded ODS B-1900 was 0.555 percent; of this only 0.124 percent was added originally as Y_2O_3. The extra oxygen in mechanically alloyed products is derived primarily from the attritor milling process, in which measured amounts of oxygen are added to control powder welding and fracture. As noted in reference 9, in milling with a grinding aid (e.g., oxygen) present, a dynamic equilibrium is reached in which grinding and welding occur while the average particle size remains constant. Reference 10 suggests that a composite powder containing a distribution of fine oxide suitable for dispersion strengthening could be obtained by using a process of grinding and welding in a nonreactive medium. From consideration of relative oxide stabilities at 1100° C, the excess oxygen may be assumed to have formed aluminum oxide (Al_2O_3) by reaction with aluminum in the alloy. This would yield, in addition to the intentional 1 volume percent of Y_2O_3, almost 2 volume percent of Al_2O_3. Reference 11 reports that, when Al_2O_3 and Y_2O_3 are present in the same alloy, the two interact to form a compound oxide. Confirming this, X-ray diffraction analysis of the residue extracted from ODS B-1900 indicated the presence of $YAlO_3$ (ASTM powder data card 16-219) and the absence of both Y_2O_3 and Al_2O_3. By spectrographic analysis, aluminum and yttrium were found in both the fine and coarse fractions of the separated extraction residue. This would indicate that some of the approximately 3 volume percent total of oxide dispersoid was coarse.

In addition to oxygen, much smaller but still significant amounts of carbon and nitrogen (table I) were detected by chemical analysis. The carbon was primarily derived from the carbon content of the raw materials and of the steel grinding balls used in the study, while the nitrogen was picked up during the processing of the powder. X-ray dif-

fraction of the extraction residue after separation into fine and coarse fractions by centrifuging indicated that titanium carbonitrides were present and that the titanium carbonitride particles segregated preferentially into the coarse fraction. Spectrographic analysis also indicated a greater titanium concentration in the coarse fraction. The incidence of titanium carbonitride particles in mechanically alloyed ODS alloys is reported in reference 12. In this instance, a calculation based on the carbon and nitrogen contents and the densities of titanium nitride and titanium carbide indicated the presence of 0.75 volume percent of these nonmetallic particles in addition to the approximately 3 volume percent of oxides.

The total volume fraction of hard phases was thus almost 4 percent. While most of the particles by number were fine and well suited to oxide dispersion strengthening, a major fraction by volume were coarse and were considered impurity phases. Such impurity particles have long been suspected of being crack initiators and contributing to low ductility in ODS materials (refs. 13 and 14). Despite the presence of some large impurity particles, the dispersion obtained in this study was comparable in oxide size, spacing, and uniformity with others which were effective for dispersion strengthening (ref. 2).

Grain morphology. - In figure 3 longitudinal and transverse views of recrystallized ODS B-1900 are shown. As may be noted from the photographs, materials with two values of GAR were produced; the first (figs. 3(a) and (b)) had a GAR of 5.5, and the second (figs. 3(c) and (d)) a GAR of 3. The average grain length and width of GAR-5.5 ODS B-1900 were 370 and 67 micrometers, while the average grain length and width of GAR-3 ODS B-1900 were 200 and 67 micrometers.

The structures achieved in this preliminary study, while definitely representing coarse and elongated grains, were far from optimum in comparison with structures which have been achieved in development programs in which the thermomechanical processing was systematically varied. For example, GAR's greater than 10 have been reported for mechanically alloyed ODS materials which were zone annealed (ref. 3). Nonetheless, achievement of large elongated grains in ODS B-1900 by conventional annealing demonstrates the potential for dispersion strengthening by mechanical alloying of this complex alloy, and, by inference, the same potential for other complex alloys normally used as cast gas turbine blades.

The achievement of recrystallization to large grains has been sought in other alloys prepared by conventional powder metallurgical techniques. In some alloys recrystallization is achieved with great difficulty if at all. Highly alloyed compositions in particular present a problem (refs. 15 to 17). However, like B-1900, other complex alloys, including some previously resistant to grain growth, might be successfully recrystallized if the mechanical alloying process were applied.

Tensile properties. - The results of tensile tests of smooth and notched specimens of ODS B-1900 are listed in table II (individual tests) and shown graphically (averages where possible) in figures 4 and 5. Table II and figure 4 include comparable smooth specimen data for cast B-1900 from reference 8.

From table II and figure 4 it may be noted that the yield strength of ODS B-1900 exceeded that of cast B-1900 at test temperatures ranging from room temperature to 1095° C. The ultimate tensile strength (UTS) of ODS B-1900 exceeded that of cast B-1900 at room temperature and 760° C, while cast B-1900 had the advantage at 870°, 980°, and 1095° C. The tensile elongation of ODS B-1900 was generally slight but rose to 4 percent at 760° C, a temperature at which many conventional superalloys show a minimum. It is interesting that the smooth specimen tensile properties were independent of grain aspect ratio. Introduction of a notch, however, increased the UTS of ODS B-1900 samples of higher GAR while decreasing the UTS of lower GAR specimens (table II and fig. 5).

At 760° C (fig. 6) and room temperature tensile fracture was transgranular (as judged by the absence of cracks at boundaries transverse to the applied stress), while at 1095° C (fig. 7) and 980° C tensile fracture was intergranular. In specimens tested at 1095° C (fig. 7) numerous cracks were noted at grain boundaries which were transverse to the applied stress. It may be surmised that the 980° and 1095° C tensile properties would be improved by elimination of transverse grain boundaries.

Stress-rupture properties. - Stress-rupture data (from individual tests) for smooth and notched specimens of ODS B-1900 with GAR's of 5.5 and 3 tested at 760° and 1095° C are listed in table III and displayed graphically in figures 8 to 11. Comparable data for smooth specimens of cast B-1900 from reference 8 are included in table III and in figures 8 and 9. Both grain aspect ratio and notch-induced stress concentration affected the stress-rupture life of ODS B-1900.

At 760° C the stress-rupture life of ODS B-1900 of both grain aspect ratios was below that of cast B-1900 (table III and fig. 8). Increasing the GAR from 3 to 5.5 doubled the 760° C stress-rupture life. At 1095° C the increase in stress-rupture life with increase in aspect ratio was even more marked, and the GAR-5.5 ODS B-1900 was somewhat superior to cast B-1900 (table III and fig. 9).

The observation of greatly increased stress-rupture life at both 760° and 1095° C with increased GAR is especially significant. The higher GAR of this study, 5.5, is less than half the GAR reported in studies in which thermomechanical processing was varied (ref. 3). This, coupled with the demonstrated 1095° C rupture life superiority of GAR-5.5 ODS B-1900, indicates the excellent potential of ODS B-1900 for providing increased high-temperature capability compared with the cast version. To realize this

potential the GAR of ODS B-1900 must be increased to levels already observed in other alloys.

Introduction of notch-induced stress concentrations greatly decreased the 760° C stress-rupture life of ODS B-1900 (table III and fig. 10). The lower GAR material was more seriously affected. At 1095° C only the lower GAR material was tested (table III and fig. 11); the presence of a notch did not decrease stress-rupture life.

At both 760° C (fig. 12(a)) and 1095° C (fig. 12(b)) stress-rupture failure for both GAR's was intergranular. The role of grain boundaries transverse to the specimen axis in providing easy fracture paths was obvious from examination of fractured specimens (fig. 12). Although B-1900 is a highly alloyed superalloy, when it was oxide dispersion strengthened, its mechanical response was quite similar to that of much less complex alloys. The same dependence on grain aspect ratio (ref. 18) and the same general fracture mode (ref. 19) had been observed in the less complex alloys. Also, as for the less complex alloys, the tensile and stress-rupture ductilities were low compared with desired levels for application as gas turbine blades. It has been shown in other alloys (ref. 3) that with reduced incidence of transverse grain boundaries both rupture elongation and reduction in area may be improved.

CONCLUDING REMARKS

The most important result of this work was demonstration of the potential for application of the mechanical alloying process to B-1900, and by inference, to other complex alloys normally used as cast gas turbine blades. In this study, both a fine and uniform oxide dispersion and recrystallization to large elongated grains were achieved in the alloy B-1900. Although the potential was thus demonstrated, optimization is yet required. The process will have to be adjusted as it was for other alloys already optimized to achieve the best results.

The need for optimization, especially of grain aspect ratio, was made graphic by the appearance of stress-rupture failures featuring numerous cracks at grain boundaries transverse to the applied stress. The benefit of even a small increase in grain aspect ratio was evident in stress-rupture life at both 760° and 1095° C. The higher grain aspect ratio material of this study also exhibited superior properties in the presence of stress concentrations in both tension and stress-rupture. While the maximum grain aspect ratio observed in this study was 5.5, grain aspect ratios of 15 or more have been obtained by varying thermomechanical processing in other studies. Zone annealing has been particularly helpful in achieving large elongated grains and should be applied to complex alloys such as B-1900. With optimization of grain aspect ratio, it is believed that ODS B-1900 could match the 760° C stress-rupture life of cast B-1900 and

show marked superiority at 1095° C. Another opportunity for increasing stress-rupture life would be to increase the added volume fraction of Y_2O_3 dispersoid, which for this study was constant at 1 percent.

It is interesting that, although ODS B-1900 contains more than 60 volume percent γ', the mechanical response of this alloy was similar to that of less complex ODS alloys containing little or no γ'; in particular, the same sensitivity to grain aspect ratio and the same fracture modes were observed. Also, as for the less complex alloys, the tensile and rupture ductilities of ODS B-1900 were low from a designer's point of view. It is anticipated that, as has been shown with less complex ODS alloys, with reduced incidence of transverse grain boundaries, the ductility would be improved. Another possible opportunity to increase ductility would be to change attritor processing conditions, particularly the atmosphere during milling, to avoid the formation of impurity phases, including excess oxides, nitrides, and carbides.

It is often difficult to achieve grain growth in nickel-base alloys prepared by powder metallurgical techniques. Highly alloyed compositions in particular present a problem. However, application of the mechanical alloying process to a complex nickel-base superalloy in this study allowed the formation of very large grains. This suggests that the process should be tried with other alloys in which recrystallization or grain growth can be achieved only with great difficulty or not at all by other means.

SUMMARY OF RESULTS

Experimental oxide-dispersion-strengthened (ODS) B-1900 to which 1 volume percent of yttrium oxide was intentionally added was prepared by the mechanical alloying process and evaluated in terms of microstructure and mechanical properties. Tensile and stress-rupture tests were performed on both smooth and notched specimens. Comparisons were made with literature data for cast B-1900. The major results of this work were as follows:

1. The microstructure of ODS B-1900 featured fine and well dispersed oxide particles present in both the γ and γ' phases. Some coarse particles, both oxides and carbonitrides, were also observed. The total fraction of nonmetallic particles was approximately 4 volume percent. After a heat treatment of 1/2 hour at 1245° C followed by 24 hours at 845° C, the grains were large and elongated. However, optimum values (> 10) of grain aspect ratio (GAR) were not achieved; measured GAR's were 3 and 5.5, well below those of optimized ODS products.

2. *The yield strength of* ODS B-1900 *exceeded that of cast* B-1900 *from room temperature to* 1095° C. The ultimate tensile strength (UTS) of ODS B-1900 exceeded cast B-1900 values at room temperature and 760° C, while at 870°, 980°, and 1095° C cast

B-1900 was superior. The tensile elongation of ODS B-1900 was generally lower than that of cast B-1900. Unnotched specimen tensile properties were generally independent of grain aspect ratio. However, in the presence of notch-induced stress concentrations the UTS of notched GAR-5.5 ODS B-1900 was higher than the UTS of unnotched ODS B-1900 at 760° C, while that of notched GAR-3 ODS B-1900 was lower. Tensile fractures were transgranular at low and intermediate temperatures and intergranular at elevated temperatures.

3. The stress-rupture life of ODS B-1900 at 760° C was below that of cast B-1900. Increasing GAR improved 760° C stress-rupture life. The presence of a notch decreased 760° rupture life, with lower GAR material more seriously affected. The stress-rupture life of ODS B-1900 at 1095° C was comparable with that of cast B-1900, with the GAR-5.5 ODS B-1900 being superior and the GAR-3 inferior to cast B-1900. Notch-induced stress concentrations did not decrease the 1095° C stress-rupture life, but did decrease the 760° C rupture life. At both 760° and 1095° C, stress-rupture fractures were intergranular and cracks were evident at transverse grain boundaries throughout the test section.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, July 28, 1975,
505-01.

REFERENCES

1. Benjamin, J. S.: Composite Metal Powder. U.S. Patent 3,591,362, July 1971.
2. Benjamin, John S.: Dispersion Strengthened Superalloys by Mechanical Alloying. Met. Trans., vol. 1, Oct. 1970, pp. 2943-2951.
3. Cairns, R. L.; Curwick, L. R.; and Benjamin, J. S.: Grain Growth in Dispersion Strengthened Superalloys by Moving Zone Heat Treatments. Met. Trans., vol. 6a, no. 1, Jan. 1975, pp. 179-188.
4. Dalal, R. P.; and Fiedler, L. J.: Effect of Superalloy Composition on Marine Environmental Resistance. Presented at the Conf. on Gas Turbine Materials in Marine Environments, Castine, Maine, July 1974.
5. Kriege, Owen H.; and Baris, J. H.: The Chemical Partitioning of Elements in Gamma Prime Separated From Precipitation-Hardened, High Temperature Nickel-Base Alloys. ASM Trans., vol. 62, no. 1, Mar. 1969, pp. 192-200.

6. Benjamin, J. S.: Dispersion Strengthened Superalloys. U.S. Patent 3,776,704, Dec. 1973.

7. Benjamin, John S.; Cairns, Robert L.; and Weber, John H.: Hot Working of Dispersion-Strengthened Heat-Resistant Alloys and the Product Thereof. U.S. Patent 3,749,612, July 1973. (Abstracted as French Patent 2,101,536, entitled Heat-Resistant, Age Hardenable Nickel Alloy Worked While Hot.)

8. High Temperature High Strength Nickel Base Alloys. 2nd Edition. The International Nickel Company, Inc., 1968.

9. Quatinetz, Max; Schafer, Robert J.; and Smeal, Charles R.: The Production of Submicron Metal Powders by Ball Milling With Grinding Aids. NASA TN D-879, 1962.

10. Arias, Alan: The Role of Chemical Reactions in the Mechanism of Comminution of Ductile Metals Into Ultrafine Powders by Grinding. NASA TN D-4862, 1968.

11. Benjamin, J. S.; Volin, T. E.; and Weber, J. H.: Dispersoids in Mechanically Alloyed Superalloys. Paper 21, Eighth Intern. Plansee Seminar, Metallwerk Plansee AG, 1974.

12. Benjamin, J. S.; and Bomford, M. J.: Effect of Yttrium Oxide Volume Fraction and Particle Size on Elevated Temperature Strength of a Dispersion Strengthened Superalloy. Met. Trans., vol. 5, no. 3, Mar. 1974, pp. 615-621.

13. Weeton, John W.; and Quatinetz, Max: Cleaning and Stabilization of Dispersion Strengthened Materials. Conf. on Oxide Dispersion Strengthening, Am. Inst. of Mining, Metallurgical, and Petroleum Engrs., 1966.

14. Hoffman, Charles A.; and Weeton, John W.: Metallographic Study of Dispersion-Strengthened Alloys After Failure in Stress Rupture. NASA TN D-3527, 1966.

15. Freche, John C.; Waters, William J.; and Ashbrook, Richard L.: Evaluation of Two Nickel-Base Alloys, Alloy 713C and NASA TAZ-8A, Produced by Extrusion of Prealloyed Powders. NASA TN D-5248, 1969.

16. Freche, John C.; Ashbrook, Richard L.; and Waters, William J.: Evaluation of a Cobalt-Base Alloy, HS-31, Made by Extrusion of Prealloyed Powders. NASA TN D-6072, 1970.

17. Freche, John C.; Ashbrook, Richard L.; and Waters, William J.: Application of Powder Metallurgy to an Advanced-Temperature Nickel-Base Alloy, NASA-TRW VI-A. NASA TN D-6560, 1971.

18. Wilcox, B. A.; and Clauer, A. H.: Dispersion Strengthening. The Superalloys. Chester T. Sims and William C. Hagel, eds., John Wiley & Sons, 1972, pp. 197-230.

19. Whittenberger, John D.: Diffusional Creep and Creep-Degradation in Dispersion-Strengthened Ni Cr Base Alloys. Met. Trans., vol. 4, no. 6, June 1973, pp. 1475-1483.

TABLE I. - COMPOSITION OF EXPERIMENTAL

ODS B-1900 AND OF CAST B-1900

Element	ODS B-1900	Cast B-1900 (nominal, ref. 8)
	Concentration, wt.%	
Carbon	0.045	0.10
Chromium	8.3	8.
Cobalt	10.2	10.
Molybdenum	5.4	6.
Aluminum	6.8	6.
Titanium	1.2	1.
Tantalum	4.2	4.
Zirconium	.08	.10
Boron	.013	.015
Yttrium oxide dispersoid	.58	-----
Total oxygen	.555	-----
Nitrogen	.056	-----
Nickel	Bal.	Bal.

TABLE II. - RESULTS OF TENSILE TESTS OF SMOOTH AND NOTCHED SPECIMENS OF ODS B-1900 WITH TWO GRAIN ASPECT RATIOS AND COMPARABLE DATA FOR CAST B-1900

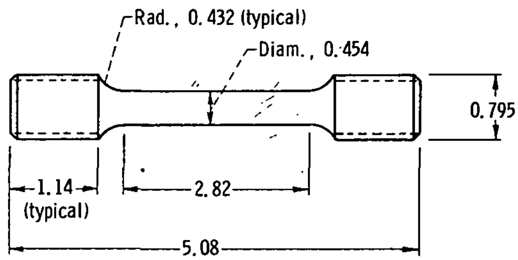
Test temperature, °C	Grain aspect ratio	Smooth or notched specimen (a)	0.2-Percent offset yield strength, MN/m²	Ultimate tensile strength, MN/m²	Elongation, percent	Reduction in area, percent
ODS B-1900						
Room temperature	5.5	S	1160	1262	1.9	4.5
	3	S	1178	1267	2.1	4.7
760	5.5	S	1069	1077	4.8	6.2
	3	S	1090	1100	3.4	5.7
	5.5	N	(b)	1221	(b)	(b)
	5.5	N	(b)	1262	(b)	(b)
	3	N	(b)	998	(b)	(b)
	3	N	(b)	995	(b)	(b)
870	3	S	721	724	1.6	3.3
980	5.5	S	453	463	1.5	3.3
	3	S	411	411	1.5	2.5
1095	5.5	S	223	223	1.6	2.5
	3	S	246	248	1.9	2.9
Cast B-1900 (ref. 8)						
Room temperature	---	S	829	973	8	(c)
760	---	S	806	952	4	(c)
870	---	S	696	793	4	(c)
980	---	S	414	552	7	(c)
1095	---	S	193	269	11	(c)

[a]Smooth, S; notched, N (stress concentration factor, 3).
[b]Not meaningful.
[c]Not available.

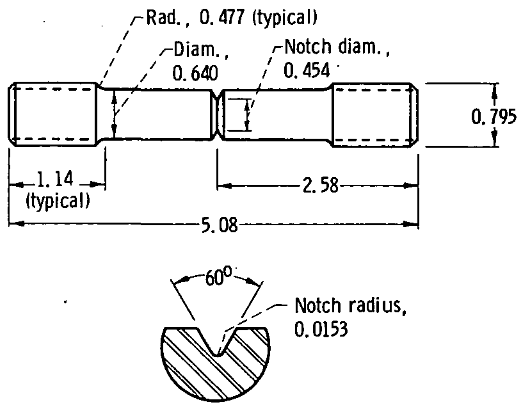
TABLE III. - RESULTS OF 760° AND 1095° C STRESS-RUPTURE TESTS OF SMOOTH AND NOTCHED SPECIMENS OF ODS B-1900 AND COMPARABLE DATA FOR CAST B-1900

Temperature, °C	Grain aspect ratio	Smooth or notched specimen (a)	Stress, MN/m^2	Rupture time, hr	Elongation, percent	Reduction in area, percent
ODS B-1900						
760	5.5	S	552	224	1.8	2.8
	3	S		98	1.5	3.6
	5.5	N		174	(b)	(b)
	3	N		11	(b)	(b)
760	5.5	S	517	396	1.3	2.1
	3	S		186	.9	2.0
	3	S		177	1.2	3.0
	5.5	N		140	(b)	(b)
	3	N		13	(b)	(b)
1095	5.5	S	68.9	143	4.1	3.9
	3	S	68.9	9	1.4	2.5
1095	5.5	S	62.1	145	1.5	2.1
	3	S		22	2.4	2.9
	3	S		41	3.5	3.6
	3	N		30	(b)	(b)
1095	3	S	55.2	44	2.8	3.7
	3	S	55.2	38	2.0	3.3
	3	N	55.2	80	(b)	(b)
Cast B-1900 (ref. 8)						
[c]760	---	S	621	100	(d)	(d)
	---	S	517	1000	(d)	(d)
1095	---	S	62.1	100	(d)	(d)
	---	S	33.8	1000	(d)	(d)

[a]Smooth, S; notched, N (stress concentration factor, 3).
[b]Not meaningful.
[c]Data extrapolated from higher temperature data.
[d]Not available.



(a) Smooth specimen.



(b) Notched specimen; stress concentration factor, approximately 3.

Figure 1. - Smooth and notched specimens used for tensile and stress-rupture tests. (All dimensions in centimeters.)

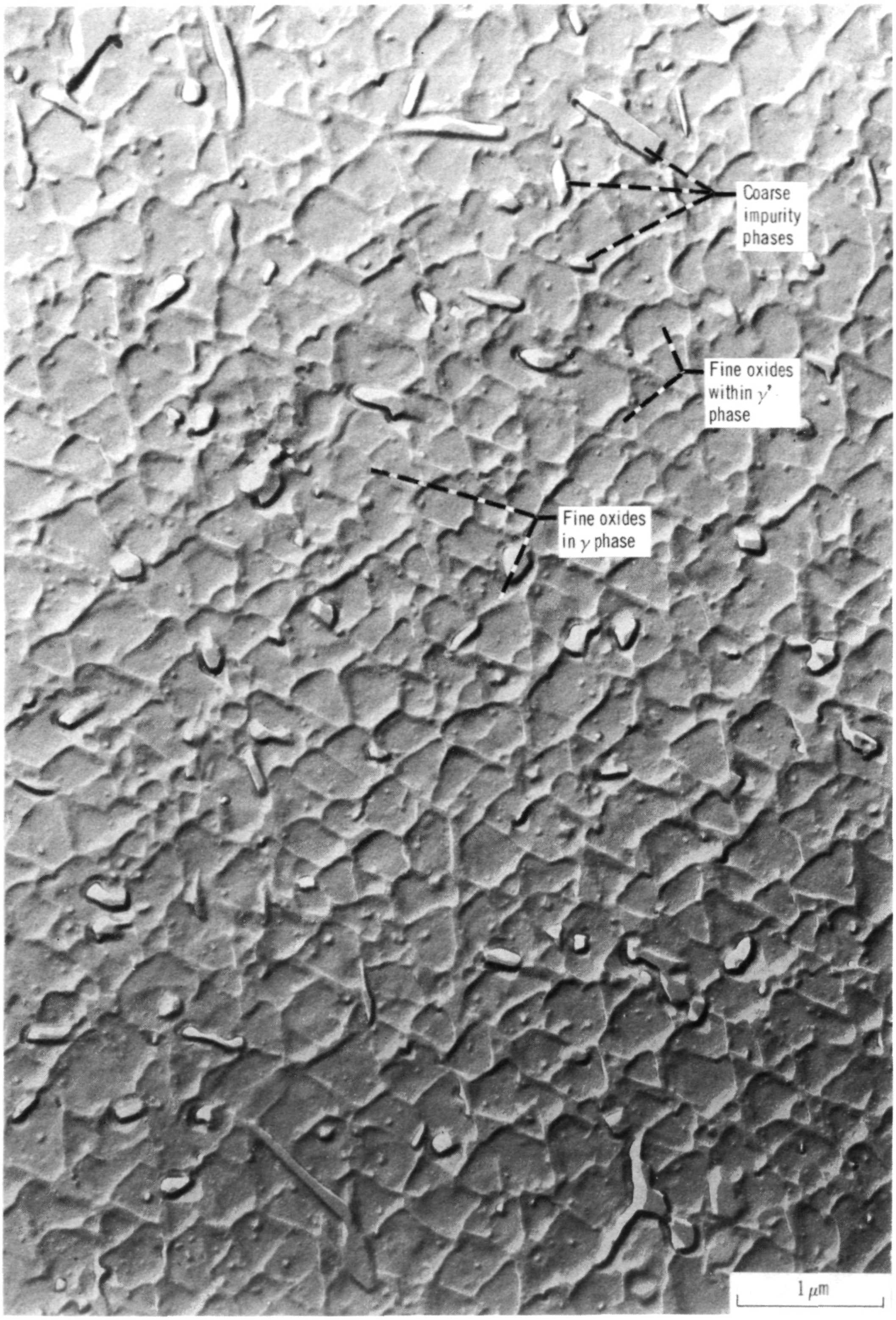
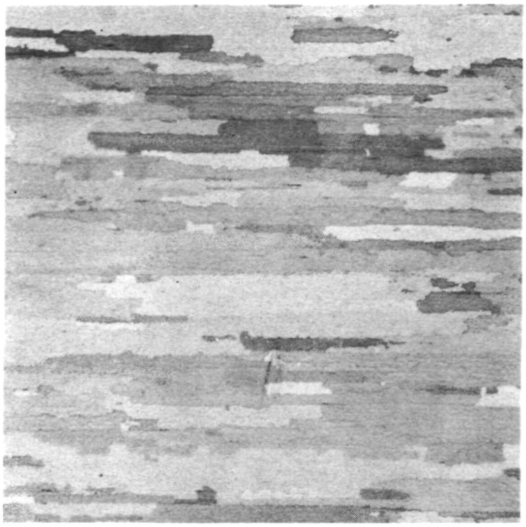
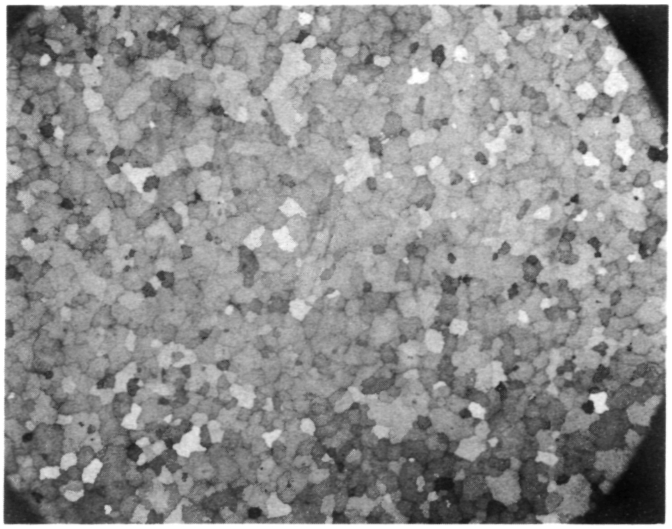


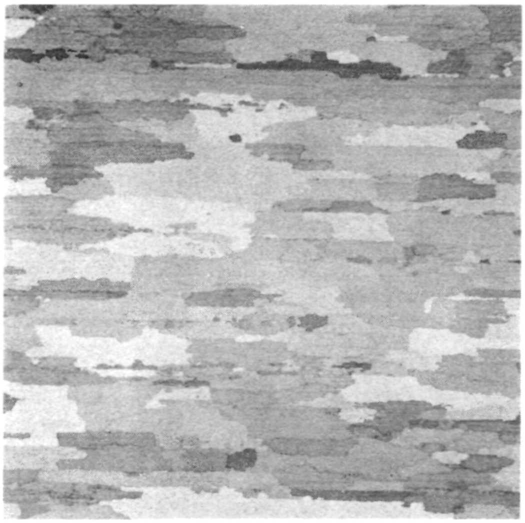
Figure 2. - Oxide-dispersion-strengthened B-1900. Note high volume fraction of γ' phase (triangular appearance), presence of both fine (0.02-μm; 200-Å) and coarse (0.5-μm; 5000-Å) nonmetallic particles, and presence of fine particles within both γ and γ' phases.



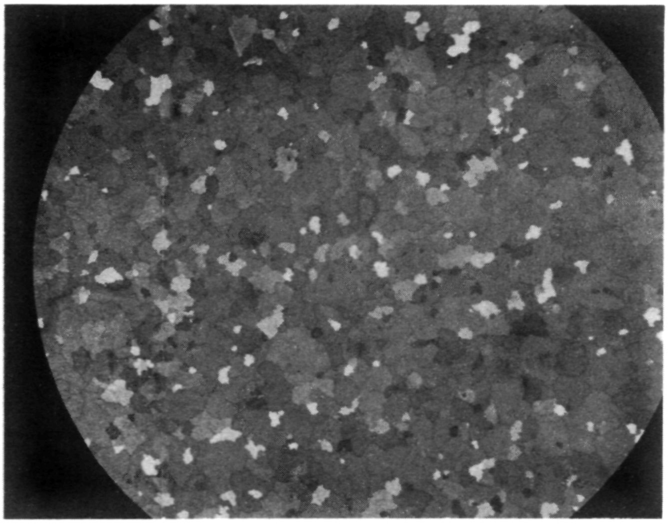
(a) GAR, 5.5; longitudinal view.



(b) GAR, 5.5; transverse view.



(c) GAR, 3; longitudinal view.



(d) GAR, 3; transverse view.

Figure 3. - Longitudinal and transverse views of ODS B-1900 with grain aspect ratios of 5.5 and 3.

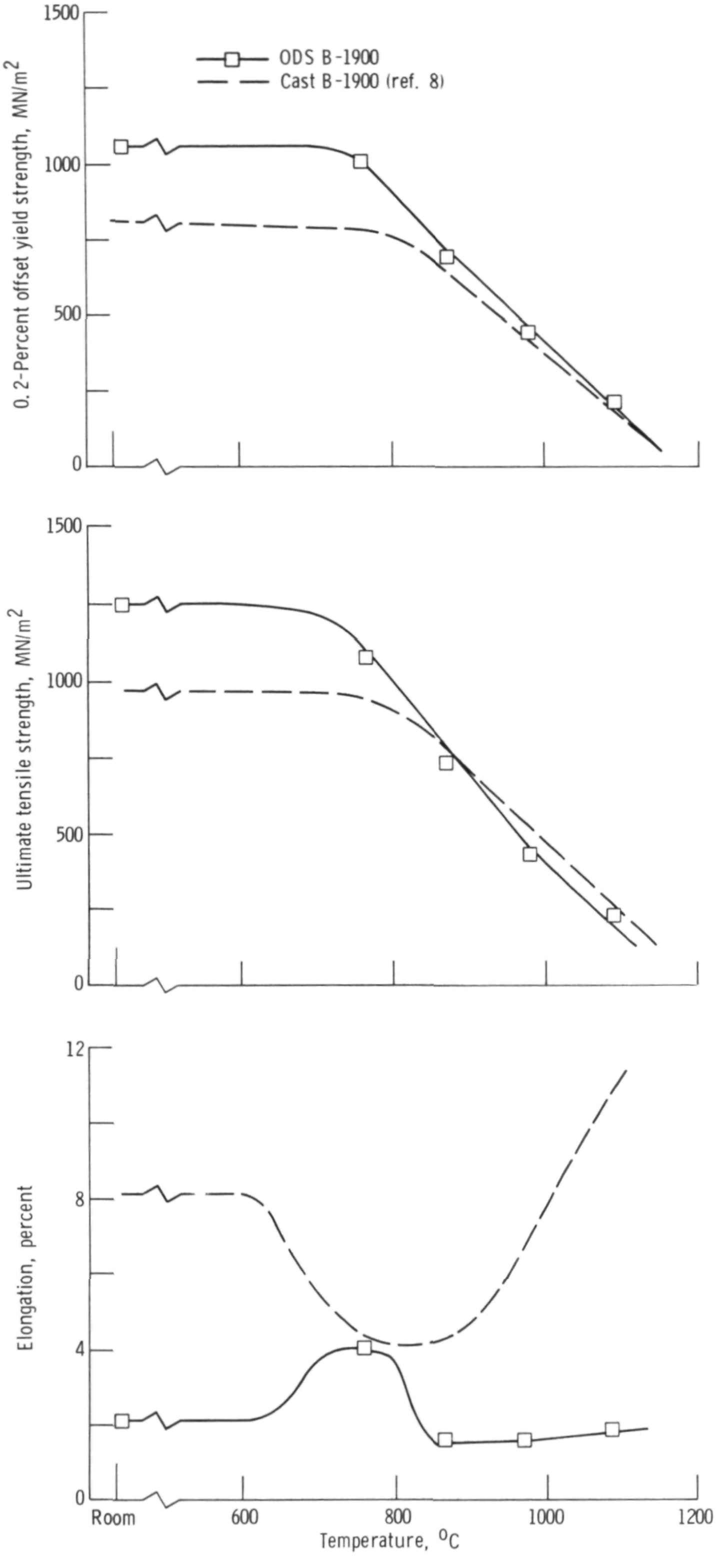


Figure 4. - Yield strength, tensile strength, and tensile elongations of smooth bar ODS B-1900 and cast B-1900.

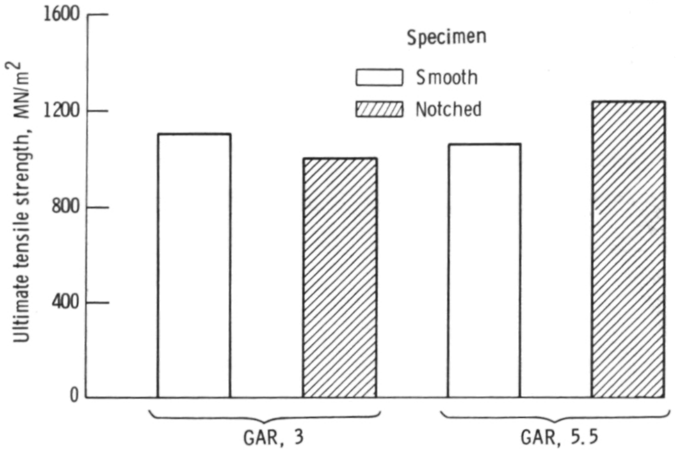
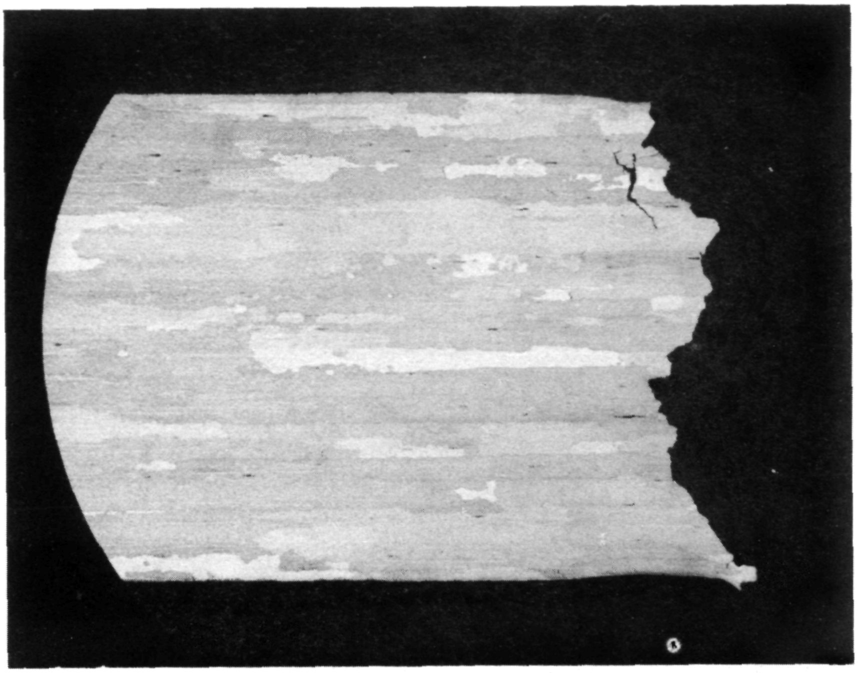
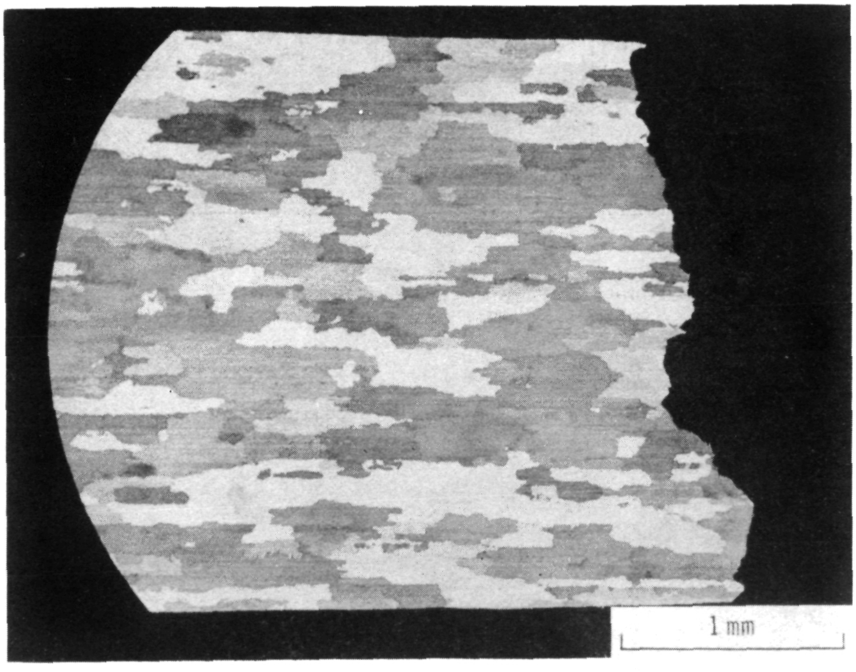


Figure 5. - Ultimate tensile strength of smooth and notched specimens of ODS B-1900 with two grain aspect ratios tested at 760° C.

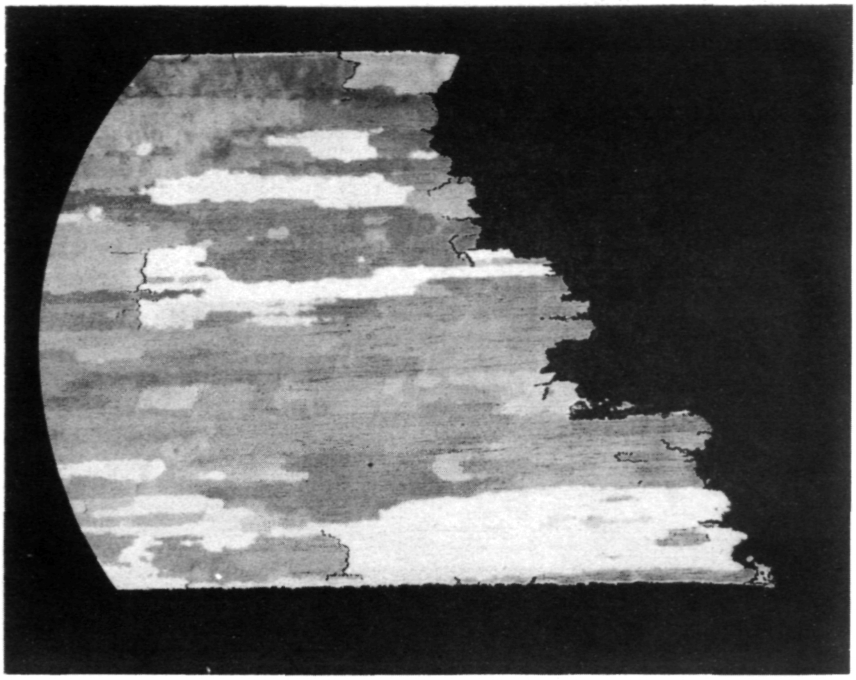


(a) GAR, 5.5; yield strength, 1069 MN/m^2; ultimate tensile strength, 1077 MN/m^2; reduction in area, 4.8 percent; note indications of necking. transgranular cracking, and absence of intergranular cracks.

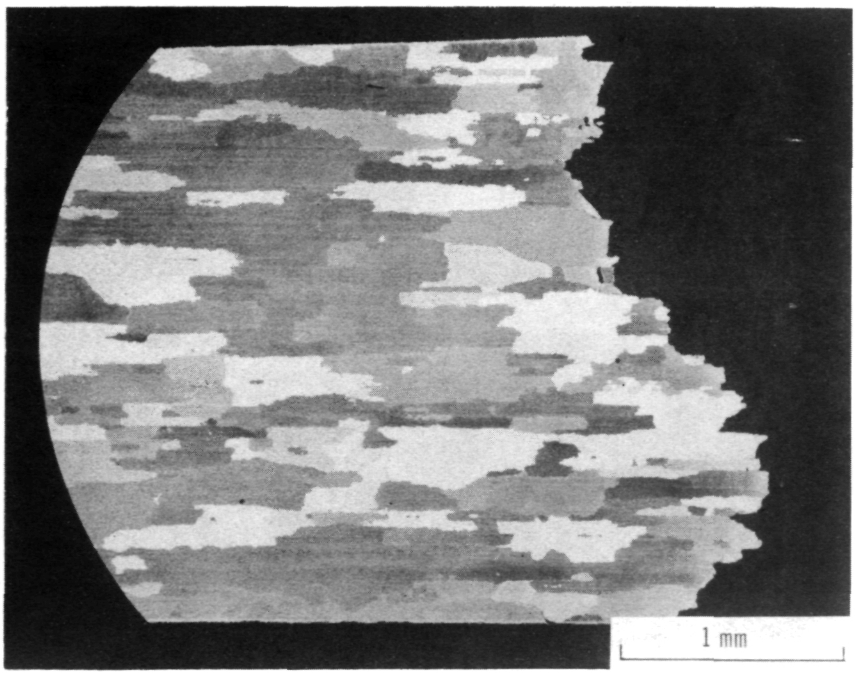


(b) GAR, 3; yield strength, 1090 MN/m^2; ultimate tensile strength, 1100 MN/m^2; reduction in area, 3.4 percent; note general absence of intergranular cracks.

Figure 6. - Fracture area of ODS B-1900 with two grain aspect ratios tested in tension at 760° C.



(a) GAR, 5.5; yield strength, 223 MN/m^2; ultimate tensile strength, 223 MN/m^2; reduction in area, 1.6 percent.



(b) GAR, 3; yield strength, 246 MN/m^2; ultimate tensile strength, 248 MN/m^2; reduction in area, 1.9 percent.

Figure 7. - Fracture areas of ODS B-1900 with two grain aspect ratios tested in tension at 1095° C.

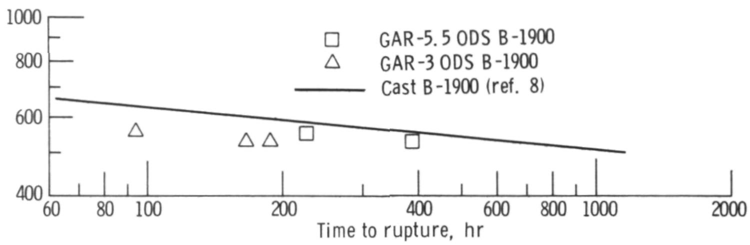


Figure 8. - Stress-rupture life at 760° C of smooth specimens of ODS B-1900 with two aspect ratios and comparable data for cast B-1900.

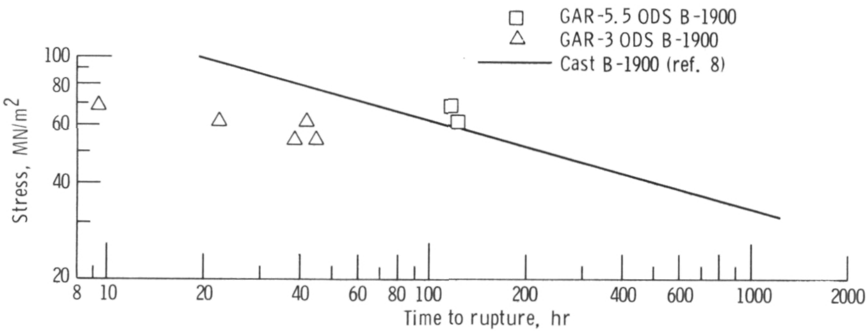


Figure 9. - Stress-rupture life at 1095° C of smooth specimens of ODS B-1900 with two aspect ratios and comparable data for cast B-1900.

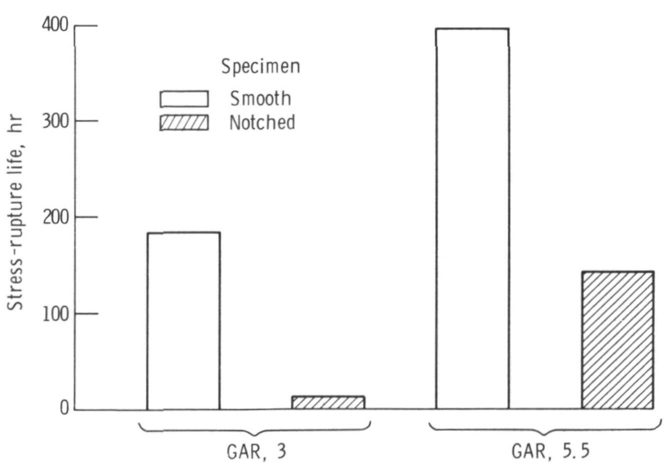


Figure 10. - Stress-rupture life at 760° C and 517 MN/m² of smooth and notched specimens of ODS B-1900 with two grain aspect ratios.

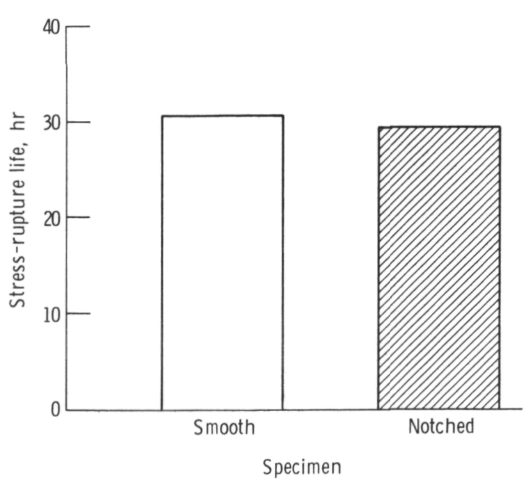
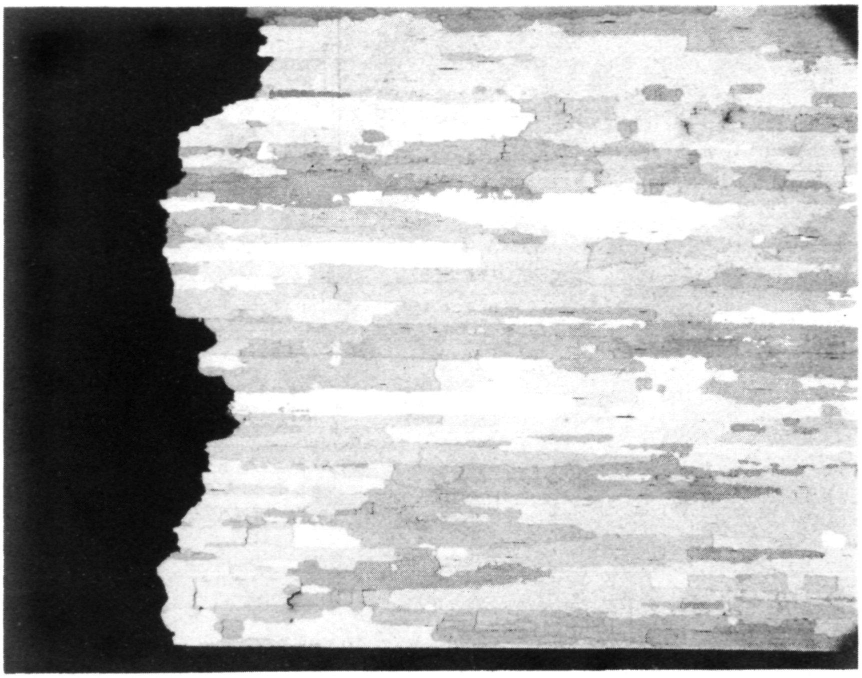
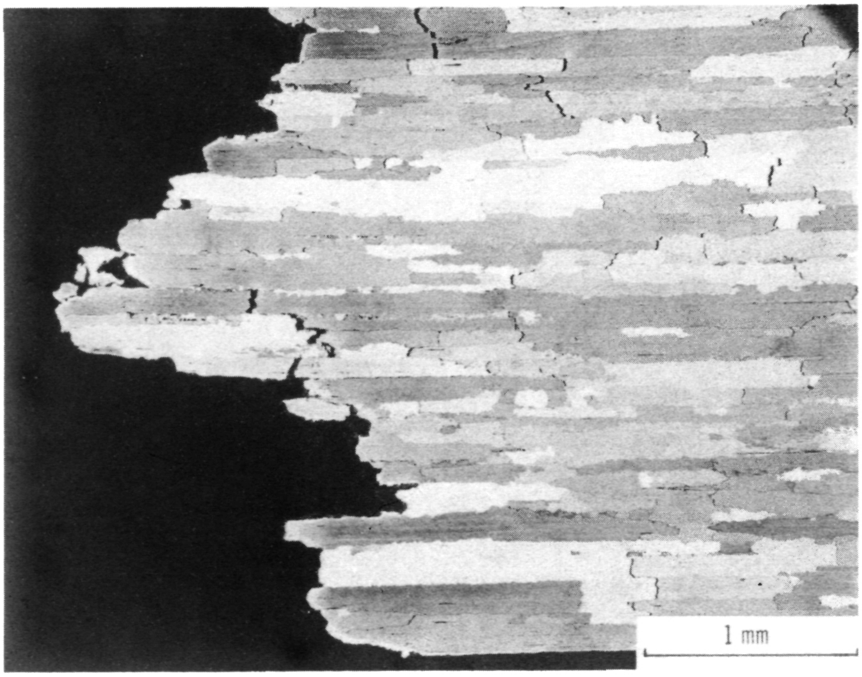


Figure 11. - Stress-rupture life at 1095° C and 62 MN/m² of GAR-3 ODS B-1900.



(a) Failure after 396 hours at 517 MN/m^2 and 760° C.



(b) Failure after 143 hours at 69 MN/m^2 and 1095° C.

Figure 12. - Fracture areas of GAR-5.5 ODS B-1900 stress-rupture tested at 760° and 1095° C.

NASA-Langley, 1975 E-8373

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